

1 TO WHOM IT MAY CONCERN:

2

3 BE IT KNOWN THAT WE, MORRIS F. DILMORE, a
4 citizen of the United States of America, residing in
5 Baker, in the County of Okaloosa, State of Florida,
6 HENRY S. MEEKS, III, a citizen of the United States of
7 America, residing in Roseville, in the County of
8 Placer, State of California, and Marc S. Fleming, a
9 citizen of the United States of America, residing in
10 Rancho Cordova, in the County of Sacramento, State of
11 California, have invented a new and useful improvement
12 in

13

14 METAL CONSOLIDATION PROCESS APPLICABLE TO FUNCTIONALLY

15 GRADIENT MATERIAL (FGM) COMPOSITIONS OF TANTALUM

16 AND OTHER MATERIALS

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1 BACKGROUND OF THE INVENTION

2 DIVISIONAL APPLICATION OF U.S. PATENT
APPLICATION SERIAL NO. 09/551,248 FILED
JUNE 13, 2000 NOW U.S. PAT. NO. 6,461,564,
3 WHICH IS

4 This application is a continuation-in-part of
5 prior U.S. patent application Serial No. 09/551,248,
6 NOW U.S. PAT. NO. 6,355,209
7 filed April 18, 2000, incorporated herein by reference.

8 This invention relates generally to the field
9 of consolidating hard metallic bodies, and also to
10 rapid and efficient heating and handling of
11 granular media employed in such consolidation, as well
12 as rapid and efficient heating and handling of preform
13 powdered metal or metal bodies to be consolidated,
14 where such bodies consist essentially of functionally
15 gradient materials, designated herein as FGM. Such
16 materials when consolidated exhibit along a body
17 dimension or dimensions decreased or varying strength
18 or ductility (strain hardening).

19 The technique of employing carbonaceous
20 particulate or grain at high temperature as pressure
21 transmitting media for producing high density metallic
22 objects is discussed at length in U.S. Patents Nos.
23 4,140,711, 4,933,140 and 4,539,175, the disclosures of
24 which are incorporated herein, by reference.

25 The present invention provides improvements
in such techniques, and particularly improvement
leading to consolidation of bodies consisting

1 essentially of functionally gradient material (FGM)
2 compositions. One example is tantalum or tantalum
3 together with other metals. Such metals, one or more
4 of which may be consolidated with tantalum, include
5 tungsten, copper, hafnium, rhenium, platinum, gold,
6 molybdenum, uranium, titanium, zirconium and aluminum.

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8 SUMMARY OF THE INVENTION

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10 It is a major object of the invention to
11 provide for consolidation of metallic powder consisting
12 of selected metals as referred to, and as may be
13 employed in target penetration, drilling, and related
14 impact activities. Such selected metals typically are
15 distributed as FGMs, as referred to.

16 It is another object of the invention to
17 provide rapid and efficient heating of carbonaceous
18 and/or ceramic particles used as pressure transmitting
19 media, and also transfer of heat generated in the
20 particles to the work, i.e. the hard metal preform to
21 be consolidated. Basic steps of the method of
22 consolidating the preform metallic body in any of
23 initially powdered, sintered, fibrous, sponge, or other
24 form capable of compaction, or densification (to reduce
25 porosity) then include the steps:

- 1 a) providing flowable particles having
- 2 carbonaceous and ceramic composition or compositions,
- 3 b) heating the particles to elevated
- 4 temperature,
- 5 c) locating the heated particles in a bed,
- 6 d) positioning the preform body at the bed,
- 7 to receive pressure transmission,
- 8 e) effecting pressurization of said bed to
- 9 cause pressure transmission via said particles to the
- 10 body, thereby to compact the body into desired shape,
- 11 as for example cylindrical shape, increasing its
- 12 density, and
- 13 f) the body consisting essentially of one
- 14 or more metals selected from the following group:
- 15 tungsten, rhenium, uranium, tantalum, platinum, copper,
- 16 gold, hafnium, molybdenum, titanium, zirconium and
- 17 aluminum,
- 18 g) the consolidated body having, along a
- 19 body dimension, one of the following characteristics:
- 20 i) decreasing strength
- 21 ii) increasing ductility
- 22 iii) decreasing strength, and increasing
- 23 ductility.
- 24
- 25
- 26

1 Another object is to achieve rapid or almost
2 instantaneous densification of a composite metal alloy
3 system, the resultant material being fine grained,
4 isotropic, and maintaining original metastable
5 microstructures.

6 A further object is to produce a consolidated
7 functionally gradient material (FGM) for use as a
8 shaped, heavy metal penetrator EFP (explosively formed
9 penetrator) or SCL (shaped charge lines) . One highly
10 advantageous and particular FGM material powder system
11 is comprised of a tantalum and other heavy metal
12 powdered alloy outer section, and transitioning to a
13 different density based powder. It may include an
14 intermediate layer of metal matrix composite of the
15 heavy metal alloy, and lower density powder, and a
16 monolithic lower density base section. The powdered
17 material system for process A may typically employ
18 tantalum particles coated with a pre-alloyed binder
19 composition but other elementally blended, mixed or
20 otherwise combined particles are applicable. The total
21 binder may typically consist of elemental metals
22 selected from the group tungsten, copper, tantalum,
23 hafnium, rhenium, platinum, gold, molybdenum, and
24 uranium hereinafter referred to as HMG, of
25 approximately 16 weight percent of the total
26 composition; but other compositions may be employed.

1 The powdered material system for a process B may
2 typically employ transition layers of one metal to the
3 next with the build-up based on requirements.

4 The ability to fabricate a functionally
5 gradient heavy metal penetrator in one single forging
6 operation has several advantages. The first is the
7 ability to design and engineer a penetrator with
8 specific and predictable dynamic performance criteria.
9 The second advantage is that of reduced manufacturing
10 costs directly related to fewer hot forging steps.
11 Additional cost reductions are realized in the area of
12 raw material usage by eliminating forging trim and
13 scrappage resulting from the use of a powder
14 metallurgy, near net shape forging preform.

15 By the use of the methodology of the present
16 invention, substantially improved structural articles
17 of manufacture can be made having minimal distortion,
18 as particularly enabled by the use of carbonaceous, or
19 ceramic, or carbonaceous/ceramic particulate in
20 flowable form.

21 An additional object includes provision of
22 a method for consolidating hard metal and/or ceramic
23 powder, and/or composite material with or without
24 polymeric powder, to form an object, that includes

25 a) pressing the FGM into a preform, and
26 preheating the preform to elevated temperature,

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1 b) providing flowable pressure transmitting
2 particles and heating said particles, and providing a
3 bed of said flowable and heated pressure transmitting
4 particles,

5 c) positioning the FGM preform in such
6 relation to the bed that the particles substantially
7 encompass the preform,

8 d) and pressurizing the bed to
9 compress said particles and cause pressure transmission
10 via the particles to the preform, thereby to
11 consolidate the preform into a desired object shape,
12 having final density.

13 The preform typically consists of tantalum complex with
14 metals selected from the HGM group as referred to.

15 An additional object is to provide a body to
16 be consolidated having varying metallic composition
17 along a body dimension. That varying composition may
18 be characterized by a series of zones, extending either
19 axially or radially for example along the article's
20 axis, each zone having a characteristic composition
21 which differs from that of an adjacent zone or zones.
22 The metal in successive zones may consist of at least
23 consolidated tantalum, and tantalum consolidated
24 together with one or more metals from the HGM group,
25 and also steel, but in varying proportions in
26 successive zones. For a projectile having great

1 penetration capability, a tapered nose zone may consist
2 primarily of tantalum, and successive zones to the rear
3 may contain less and less tantalum and more and more
4 steel.

5 For a three metal body, the metals being M_1 ,
6 M_2 and M_3 , the weights W_1 , W_2 and W_3 per unit volume of
7 the respective metals M_1 , M_2 and M_3 are related and
8 selected, to be as follows:

9
$$W_1 > W_2 > W_3$$

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11 Other objects are to provide consolidated
12 bodies such as tapered shells, and/or cylindrical and
13 tapered bodies, made by the method of the invention,
14 and having functional gradient properties in two
15 dimensions.

16 The novel features which are believed to be
17 characteristic of this invention, both as to its
18 organization and method of operation, together with
19 further objectives and advantages thereof, will be
20 better understood from the following description
21 considered in connection with the accompanying drawings
22 in which a presently preferred embodiment of the
23 invention is illustrated by way of example. It is to
24 be expressly understood, however, that the drawings are
25 for the purposes of illustration and description only

1 and are not intended as a definition of the limits of
2 the invention.

3 DRAWING DESCRIPTION

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5 Fig. 1 is a flow diagram showing method steps
6 of the present invention;

7 Fig. 2 is a cut-away elevation showing the
8 consolidation step of the present invention;

9 Fig. 3 is a vertical section showing preform
10 pressurization, prior to consolidation;

11 Fig. 4 is a view like Fig. 3, showing a
12 modified preform;

13 Fig. 5 is a view of a consolidated preform;

14 Fig. 6 shows a tantalum particle with layers
15 of Z_1 , Z_2 , and Z_3 as found in a matrix;

16 Fig. 7 is a section taken through multiple
17 layers of different metals;

18 Figs. 8a and 8b are side and bottom views of
19 a consolidated shaped charge liner (SCL) formed by the
20 method of the invention; and

21 Figs. 9a and 9b are side and bottom views of
22 a consolidated explosively formed penetration (EFP)
23 formed by the method of the invention.

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1 DETAILED DESCRIPTION

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3 Referring first to Fig. 1, there is shown a
4 flow diagram illustrating method steps of the present
5 invention. As can be seen from numeral 10, initially a
6 metal, metal and ceramic, or ceramic article of
7 manufacture or preform is made, for example, in the
8 shape of a penetrator or other body or impact tool such
9 as a drill or other product. One preferred embodiment
10 contemplates the use of a metal preform made of
11 powdered tantalum, partially coated with one or more
12 HGM particles, then mechanically blended with a low
13 alloy steel powder. Preferably, tantalum constitutes
14 more than 50% of the overall weight of the preform.
15 Other metallic or ceramic particles or coatings may
16 also be included. See for example Fig. 6 showing
17 tantalum particles 100 coated with or surrounded by
18 metals Z_1 , Z_2 , and Z_3 , in a preform. A preform
19 typically is about 60 to 85 percent of theoretical
20 density after the powder has been made and compacted
21 into a preformed shape, and it may typically
22 subsequently be sintered (see step 12 in Fig. 1) in
23 order to increase the strength. In the preferred
24 embodiment, the preform in billet form is subjected to
25 cold or ambient temperature isostatic compaction at

1 about 60,000 pounds per square inch, preferably within
 2 an evacuated and sealed elastomeric (rubber) container.
 3 See for example Fig. 3 showing evacuated, sealed
 4 elastomeric container 110, with the preform 111 located
 5 therein, and shaped in the form of a cylinder. Fig. 5
 6 is like Fig. 3, but shows the preform 112 shaped in the
 7 form of a cylinder and having a tapered end 112a, for
 8 penetration of hard targets. Fluid pressure is
 9 supplied at 113 to the interior 114 of a metal vessel
 10 115 within which the tantalum, and other powdered metal
 11 (M_1 , M_2 , etc.) preform, and its elastomeric container
 12 are located, to pressurize the container and compact
 13 the powder preform. Once the billet preform has been
 14 compacted to about 60% of theoretical density, it is
 15 heated in a protective or reducing atmosphere, such as
 16 Argon or hydrogen, to above 900°C, in preparation for
 17 consolidation. See step 14 in Fig. 1. Alternative
 18 steps include step 12 sintering in Fig. 1, and re-
 19 heating at 14.

20 The consolidation process, illustrated at 16
 21 in Fig. 1, takes place after the hot preform (removed
 22 from 110 and 115) has been placed, as for example in a
 23 bed of heated carbonaceous or carbonaceous/ceramic
 24 particles as hereinbelow discussed in greater detail.
 25 Consolidation takes place by subjecting the embedded

1 preform to elevated temperature and high pressure. In
2 a preferred embodiment, temperatures in the range of
3 about 1,600°F. and uniaxial pressures of about 5 to 100
4 and higher TSI are used, for compaction. The preform
5 has now been densified and can be separated, as noted
6 at 18 in Fig. 1, whereby the carbonaceous particles
7 separate readily from the preform and can be recycled
8 as indicated at 19. If necessary, any particles
9 adhering to the preform can be removed and the final
10 product can be further finished, as for example
11 machined.

12 Final product dimensional stability, to a
13 high and desirable degree, is obtained when the
14 particle (grain) bed primarily (and preferably
15 substantially completely) consists of flowable
16 carbonaceous and/or ceramic particles. For best
17 results, such carbonaceous particles are resiliently
18 compressible graphite beads, and they have outward
19 projecting nodules on and spaced part on their
20 generally spheroidally shaped outer surfaces, as well
21 as surface fissures. See for example U.S. Patent
22 No.4,640,711. Their preferred size is between 50 and
23 240 mesh. Useful granules are further identified as
24 desulphurized petroleum coke. Such carbon or graphite

1 particles have the following additional advantages in
2 the process:

- 3 1. They form easily around corners and
4 edges, to distribute applied pressure
5 essentially uniformly to and over the body
6 being compacted. The particles suffer very
7 minimal fracture, under compaction pressure.
- 8 2. The particles are not abrasive, therefore
9 reduced scoring and wear of the die is
10 achieved.
- 11 3. They are elastically deformable, i.e.
12 resiliently compressible under pressure and
13 at elevated temperature, the particles being
14 stable and usable up to 4,000°F.; it is found
15 that the granules, accordingly, tend to
16 separate easily from (i.e. do not adhere to)
17 the body surface when the body is removed
18 from the bed following compaction.
- 19 4. The granules do not agglomerate, i.e.
20 cling to one another, as a result of the
21 body compaction process. Accordingly, the
22 particles are readily recycled, for reuse, as
23 at 19 in Fig. 1.
- 24 5. The graphite particles become

1 rapidly heated in response to passage of
2 electrical current or microwaves
3 therethrough. The particles are stable and
4 usable at elevated temperatures up to
5 4,000°F. Even though graphite oxidizes in
6 air at temperatures over 800°F. Short
7 exposures as during heatup and cooldown, do
8 not substantially harm the graphite
9 particles.

10 Referring now to Fig. 2, the
11 consolidation step is more completely illustrated. In
12 the preferred embodiment, the preform 20 (as for
13 example preform 111 in Fig. 3 or preform 111a in Fig.
14 4) has been completely embedded in a bed of
15 carbonaceous particles 22 as described, and which in
16 turn have been placed in a contained zone 24a as in
17 consolidation die 24. Press bed 26 forms a bottom
18 platen, while hydraulic press ram 28 defines a top and
19 is used to press down onto the particles 22 which
20 distributes the applied pressure non-isostatically (30%
21 deformation (compression) axially - 10% deformation
22 (tensile) radially) to the preform 20. The preform is
23 at a temperature between 200°C. and 1,800°C., prior to
24 compaction. The embedded metal powder preform 20 is
25 rapidly compressed under high uniaxial pressure by the

1 action of ram 28 in die 24, the grain having been
 2 heated to between 400°C. and 4,000°F. Pressurization
 3 is typically effected at levels greater than about
 4 20,000 psi for a time interval of less than about 30
 5 seconds. Particles may be located within a sub-bed in
 6 a deformable container, in bed 22.

7 Referring again to Fig. 2, a heating furnace
 8 50 is shown, incorporating a fluidized bed of grain
 9 particles, to be supplied at 51 to die 24. Such PTM
 10 can be a carbonaceous and ceramic composite of varying
 11 composition ranging from 5 to 95 percent, by volume, of
 12 ceramic particles, the balance being carbonaceous
 13 particles. Usable ceramics include: aluminum oxide,
 14 boron carbide or nitride, and other hard ceramic
 15 materials. The heater may comprise an electrical
 16 resistance heater, or a microwave heater, for example.

17 Fig. 4 shows a preform 111a, similar to that
 18 at 111 in Fig. 3; however, the metal composition of the
 19 preform varies along its length direction, indicated by
 20 arrow 140. A stratified overall composition is
 21 indicated by multiple layers as for example at 142-145.
 22 Each layer may consist of one or more of powder form
 23 metals M_1 and M_2 (or mixture thereof), or metals M_1 , M_2
 24 and M_3 (or mixtures thereof), or metals M_1 , M_2 , M_3 , M_4 ,
 25 M_5 , and M_6 (or mixtures thereof). The selection of

1 metals and mixtures, and their proportions as by
2 weight, may be such as to produce an ultimate
3 consolidated article wherein the strength and ductility
4 of the article (at zones corresponding to layers 142-
5 145) varies, in the length direction 140; for example
6 the hardness may decrease, progressively, in direction
7 140.

8 In Fig. 4, each layer may consist of one or
9 more of powder form metals M_1 and M_2 (or mixture
10 thereof), or metals M_1 , M_2 and M_3 (or mixtures thereof),
11 or metals M_1 , M_2 , M_3 and M_4 (or mixtures thereof), or
12 metals M_1 , M_2 , M_3 , M_4 and M_5 (or mixtures thereof), or M_1 ,
13 M_2 , M_3 , M_4 , M_5 , and M_6 (or mixtures thereof). Again, the
14 selection of metals may be such that ultimate strength
15 decreases and ductility increases, progressively and
16 stepwise, in direction 140. Thus, for example, the
17 layer 142 consists of the very strong high density
18 metal such as tantalum adapted for high velocity
19 penetration of armor plate, or other hard target
20 structures such as reinforced concrete and steel,
21 underground bunkers such as those used to protect
22 chemical and biological weapons of mass destruction
23 (WMD). The opposite end layer 145 may consist
24 primarily of copper, etc. for high ductility and
25 performance.

1 Layer 142 may consist of particles of
2 tantalum encapsulated within layers of one or more HGM
3 metal particles, and defined as powder A. Layer 145
4 may consist of particles of low alloy steel, defined as
5 powder B. Intermediate layers 143 and 144 may consist
6 of mixtures of powder A and powder B, where the
7 percentage by weight of powder A decreases in
8 successive layers in direction 140, and the percentage
9 by weight of powder B in successive layers increases in
10 direction 140.

11 One example of the transition layer
12 composition in Fig. 4 would be as follows:

13 Layer 142 consists primarily of powder A

14 Layer 143 consists of 80% powder A and 20% powder B

15 Layer 144 consists of 60% powder A and 40% powder B

16 A further layer if used consists of 40% powder A and
17 60% powder B

18 A further layer if used consists of 20% powder A and
19 80% powder B

20 Layer 145 consists of 100% powder B

21 A further definition of the composite is as
22 follows: the body may be of a SCL or EFP shape as
23 discussed rates, the body consisting of at least two
24 metals, M_1 and M_2 , the proportions of M_1 and M_2 in said
25 body nose portion and second body portion being

1 different. For example, the metal M_1 is tantalum, the
2 proportion of tantalum in that nose portion being
3 greater than the proportion of tantalum in said second
4 body portion. Further, the body has third and fourth
5 body portions along said dimension, the proportion of
6 tantalum in said second body portion exceeding the
7 proportion of tantalum in said third body portion, and
8 the proportion of tantalum in said third body portion
9 exceeding the proportion of tantalum in said fourth
10 body portion.

11 In addition, the body has first and second
12 ends, the consolidated metal at the first end having
13 higher density than the consolidated metal at the
14 second end; and wherein the metal at the first end
15 consists primarily of tantalum, and the metal at the
16 second end consists primarily of a different density
17 and performance characteristic material, i.e.,
18 pyrophoric.

19 Fig. 5 shows by way of example a product 160
20 shaped generally like that of the preform 111a. The
21 product 160 has been pressure consolidated, as
22 described, to reduce its size from preform size
23 indicated by the broken lines 161. Forward portion 162
24 consists essentially of tantalum; the next layer
25 portion 163 in sequence consists of 20% by weight of a
26 lower density metal (LDM) and the balance tantalum; the

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1 next layered portion 164 in sequence consists of 40%
2 lower density metal (LDM) and the balance tantalum; the
3 next layered portion 165 in sequence consists of 60%
4 lower density metal and the balance tantalum; the next
5 layered portion 166 in sequence consists of 80% lower
6 density metal (LDM) and the balance tantalum; and the
7 last layer 167 consists essentially of LDM. The layer
8 thicknesses can be adjusted to lower increments to
9 improve the FGM bond.

10 The process of the invention yields a fully
11 dense microstructure and metallurgically sound bonds at
12 180-184, across the layered zones 162-167.

13 In Fig. 7 a "Process B" formed shape 120
14 consists of metallic layers 121-123 with decreasing
15 strength in direction 124. The layers are consolidated
16 as described above. Typical layers are:

17 121 - tantalum
18 122 - copper
19 123 - aluminum

20 Density decreases in direction 124.

21 In Figs. 8a and 8b, a shaped charge liner 80
22 has conical shell form, with a base 81, convex nose 82,
23 outer side wall 83 tapering toward 82, and inner side
24 wall 84 tapering toward 82. Wall 84 surrounds or forms
25 inner cavity 85. The liner is formed by the method of
26 the invention, i.e. is a consolidated body, and has FGM

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1 property (decreasing strength and/or ductility) in
2 axial length direction 87; and FGM property (decreasing
3 hardness and/or toughness) in wall thickness direction
4 88, those directions indicated by arrows, as shown.
5 Thus, the outer side is more ductile than the inner
6 side, and the nose 82 is more ductile than the base 81.

7 In Figs. 9a and 9b, a penetrator 90 has
8 combined cylindrical and tapered shape (as at sections
9 90a and 90b as shown), and is a solid body. Section
10 90b tapers toward tip 91. The penetrator is formed by
11 the method of the invention, i.e. is a consolidated
12 body, and has FGM property (increasing strength and/or
13 ductility in axial length direction 93; and FGM
14 property (decreasing strength and/or ductility) in
15 center-to-side directions 94. Those directions are
16 indicated by arrows as shown. Thus, the tip 91 and
17 tapered wall 96 are stronger than the base 98; and body
18 outer side 99 is stronger than body center 100.

19 In Figs. 10a and 10b, an EFP body 110 is
20 shown in side and bottom views. A body hollow 111 is
21 formed below a domed top 112.

22 In each of Figs. 8a, 8b, 9a, 9b, 10a, and
23 10b, the body at its toughest zone may consist of
24 tantalum, and at less tough zone may consist of
25 tantalum complexed with metal or metals selected from
26 the above HGM group.

1 The basic preferred method of consolidating a
2 body in any of initially powdered, sintered, fibrous,
3 sponge, or other form capable of compaction, that
4 includes the steps:

5 a) providing flowable pressure transmission
6 particles having carbonaceous and ceramic composition
7 or compositions,

8 b) heating said particles to elevated
9 temperature,

10 c) locating said heated particles in a bed,

11 d) positioning said body at said bed, to
12 receive pressure transmission,

13 e) effecting pressurization of said bed to
14 cause pressure transmission via said particles to said
15 body, thereby to compact and consolidate the body into
16 desired shape, increasing its density;

17 f) the body consisting essentially of one
18 or more metals selected from the following group:
19 tungsten, rhenium, uranium, tantalum, platinum, copper,
20 gold, hafnium, molybdenum, titanium, zirconium and
21 aluminum;

22 g) said consolidated body having, along a
23 body dimension, one of the following characteristics:

24 i) decreasing strength

25 ii) increasing ductility

1 iii) decreasing strength, and increasing
2 ductility.

3 Typically, the body has varying metallic
4 composition along said dimension; and the varying
5 metallic composition is characterized by a series of
6 zones, the metal of each zone having a characteristic
7 composition which differs from that of an adjacent zone
8 or zones. Further, the metals in at least two
9 successive zones consist substantially of tantalum, and
10 tantalum consolidated with a metal or metals selected
11 from the group tungsten, rhenium, uranium, tantalum,
12 platinum, copper, gold, hafnium, molybdenum, titanium,
13 zirconium and aluminum.

14 The body may consist of powders of metals
15 that have been initially combined and compressed into
16 body form, at pressure exceeding 20,000 pounds per
17 square inch, prior to said step e) pressurization. At
18 least part of the body has one of the following forms:

- 19 i) cone
20 ii) lens
21 iii) cylinder
22 iv) cylinder and cone combination
23 v) cylinder and lens combination.

24 The disclosure of U.S. Patent Application
25 Serial No. 09/239,268 is also incorporated herein, by

1 reference. Accordingly, the consolidated tantalum may
2 have $\langle 111 \rangle$ texture less than about 2.8X random.

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